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(6) THE BEHAVIOR OF SOME WAXES IN COMPOSITION B •

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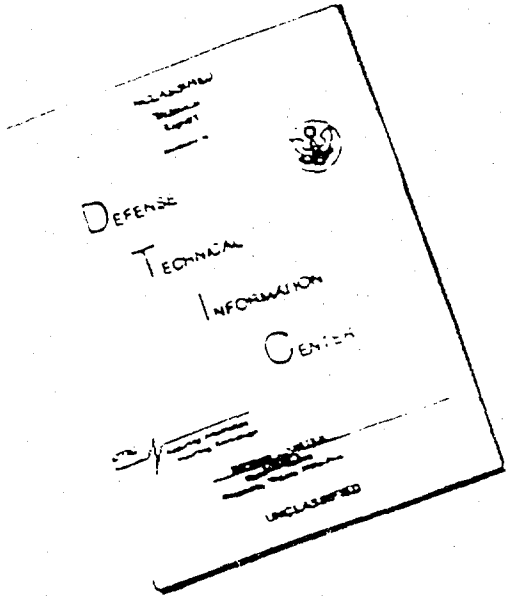

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Gel Permeation Chromatography Analysis	105 mm shell	
Mass spectrometry		

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A gel permeation chromatographic technique, checked by mass spectrometry, has been used to determine the molecular weight distributions of the various hydrocarbon waxes. The technique shows high potential as a method for determining the degree of fractionation of waxes in Comp B casts. The dispersive, wettability and solubility properties of the waxes in the TNT/RDX matrix were also investigated.

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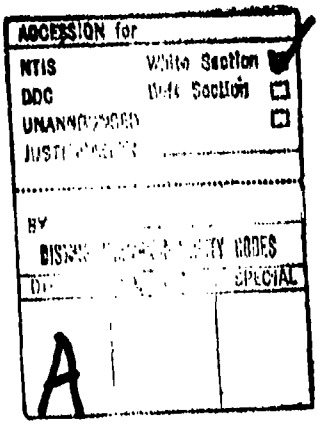

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INTRODUCTION

It was reported (ref. 1) that the addition of one percent wax to Composition B influenced its pouring qualities during formulation, and its sensitivity in the final cast state. However, not all of the waxes studied were observed to have influenced both these parameters satisfactorily. An acceptable mixture was attributed to an effective wax coating of the surface of the RDX crystals. The wax had to wet the RDX surface to cover it, and it was thought that differences in the wetting ability among the various waxes examined accounted for the differences observed in the properties of the mixture.

The question of wettability of RDX by waxes must be resolved first at ambient temperatures prior to casting. Then, the stability of adhesion of the wax coating on RDX during and after casting operations must be examined closely. Before answers to these questions can be sought, a more complete characterization of waxes is required. This report describes a preliminary investigation of the compositional analysis of some of the waxes considered for use in Comp B. Included in this study are some checks on the wettability of RDX by TNT and the waxes.

EXPERIMENTAL

Materials

The waxes investigated were: Sunoco 8810, Sunoco 985B, Sunoco 1290B, Indramic 170C, Witco 68097, Standard Wax of Knoxville 123, Petrolite ES670, and Beeswax.

Volunteer Army Ammunition Plant grade TNT and Holston Class A RDX were used in most of the experiments. In certain cases, as noted, the RDX was recrystallized from acetone prior to its use.

Bead Preparation

Small beads of wax and/or TNT were prepared by inserting the end of a clean tungsten wire to a predetermined depth into the molten substances and removing it to allow the resulting bead to solidify. The bead thus formed was then repeatedly dipped until the desired size was attained.

Preparation of RDX Surfaces

Pelletized surfaces of RDX were formed by pressing Class A RDX, recrystallized from methyl ethyl ketone, at 2109 Kg/cm^2 (30,000 psi) for one hour. Only those pellets having flat crystalline faces suitable for contact angle measurements were chosen.

Contact Angle Measurements

Contact angles of the wax and/or TNT beads on the RDX crystalline surface were measured using a telescope fitted with a goniometer eyepiece system manufactured by Rame-Hart, Inc., Mountain Lakes, NJ. Measurements were carried out at ambient atmosphere and in a water-saturated atmosphere. The temperature was controlled at 83°C by enclosing the test surface and bead in a thermostatted cell provided with viewing windows. Angles were measured on the right and left sides of the molten bead in contact with the surface, and their average reported as a single contact angle determination. Two such determinations were made for each contact angle measurement; one of the bead as prepared and a second of the droplet formed upon introduction of additional material. This procedure gave the most reproducible measurements.

Determination of Solubility of Wax in TNT

A mixture of 975 parts of TNT and 25 parts of wax was placed in a small test tube, melted and thoroughly mixed. The mixture was allowed to stand in a steam bath for one hour, undisturbed. The test tube was then removed from the bath and cooled to room temperature. In the solid TNT/Wax mixture there was a noticeable separation of the wax to the top and sides of the test tube. The central portion of the cast was isolated and weighed into a tared filtering crucible. The TNT was eluted with acetone and any separated wax was determined by reweighing the tared crucible. As a control, the solubility of the wax in acetone was tested and found to be negligible.

Mass Spectrometric Analysis of the Waxes

Mass spectra were determined at the City University of New York on a Varian MAT CH-5 single focusing mass spectrometer and on an AEI MS-902 double-focusing mass spectrometer. In both cases samples were introduced via the direct inlet system with an electron beam energy of 70 eV and a source temperature of 350°C.

Gel Permeation Chromatographic (GPC) Analysis of Waxes

Wax samples, neat and from Comp B core specimens, were analyzed for their molecular weight distributions on a Waters GPC Model 200, at Waters Associates, Milford, MA. This instrument is capable of operation up to 150°C throughout the entire system, insuring the solubility of the polymer waxes at all times during an analysis. The GPC was equipped with a bank of five 10^6 Å Styragel® columns

in series. The solvent, 1,2,4-trichlorobenzene (TCB), contained 50 ppm of scavenger for sample protection.

A typical calibration curve is shown in figure 1 which relates the molecular weight (contour chain length) of monodispersed polystyrene and hydrocarbon standards against their retention volume. The molecular weights derived from the calibration curve are based upon the chain length of the dissolved polystyrene and normal hydrocarbon standards.

The conditions utilized for the analyses were:

Solvent:	TCB
Sample concentration	1/8 to 1/2% w/v hot filtered through a Krueger filter to remove particulate matter
Columns	Styragel® 500Å, 250Å, 100Å, 60Å
Flow rate	1.0 ml/min
RI attenuation	1X
Chart speed	6 in/hr
Temperature	135°C
Injection size	2 cc
Calibration curve	C-1065

Wax Distribution in Comp B Determination

GPC/MS Determination

Core samples, 12.7 mm diameter x 25.4 mm length (½" x 1") were taken from the Comp B cast contained in the two 105 mm projectiles. Comp B cast K-254 formulated with Petrolite ES670 was loaded at Kansas Army Ammunition Plant (KAAP) and Comp B cast J-212 formulated with Indramic 170-C was loaded at Joliet Army Ammunition Plant (JAAP). The instrument chosen for the GPC analysis of the wax content in the core samples was the Waters Model 200. The conditions utilized were:

Sample concentration:	5%
Injection size:	2 ml
RI attenuation:	1X
Solvent:	1,2,4-trichlorobenzene
Flow rate:	1.0 ml/min
Analysis time:	4 hours
Chart spec:	6 in/hr
Temperature:	135°C
Columns:	Styragel® 10^6Å, 10^5Å, 10^4Å 10^3Å, 500Å
Calibration curve:	C-1055

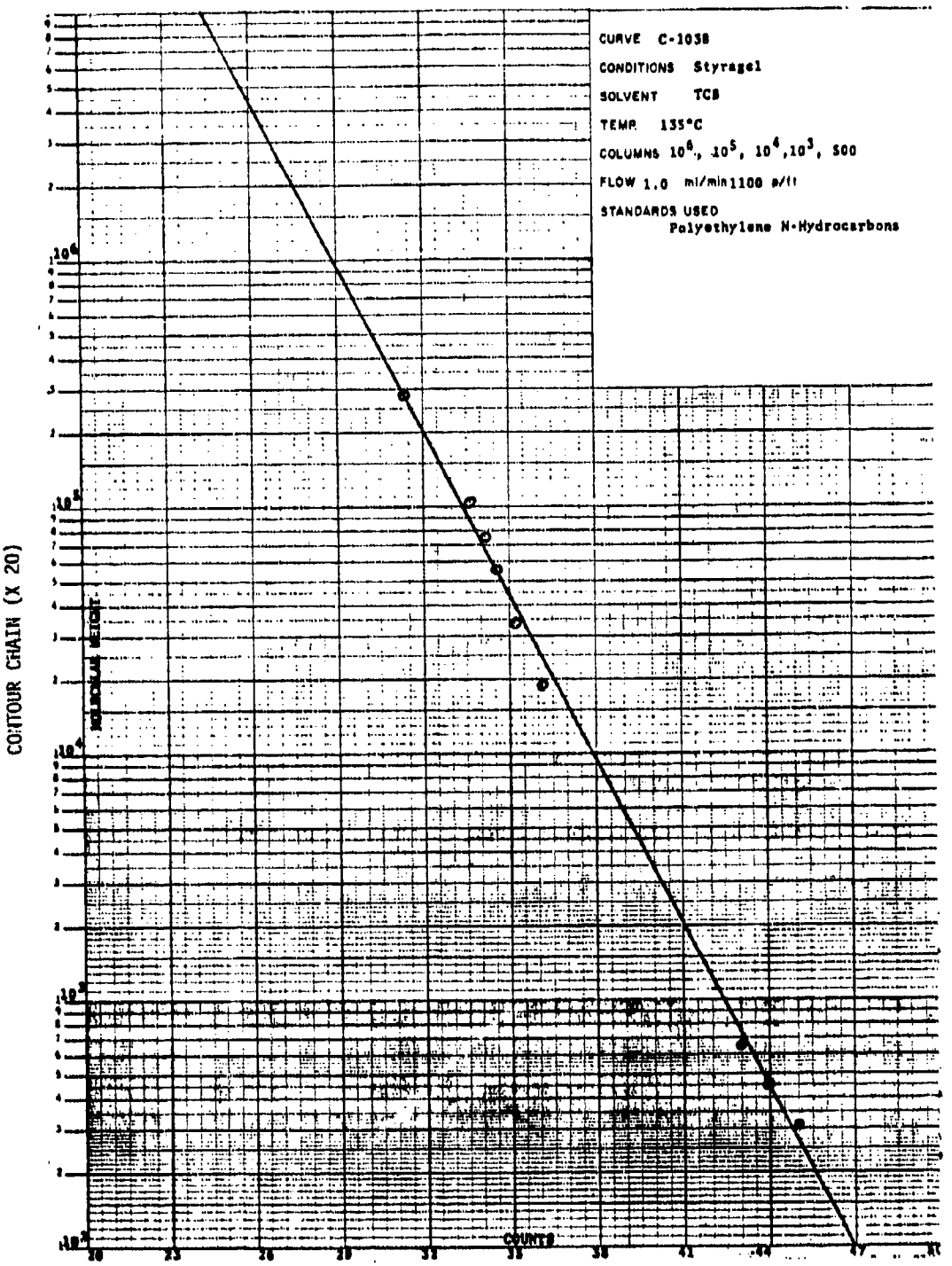

Figure 1. Gel permeation chromatography calibration curve.

Each of the waxes eluted between the 30th and 45th counts on the chromatogram. Any components eluting after these counts were attributable to RDX and TNT and not treated.

Aliquots of the same core samples analyzed by GPC were subjected to mass spectral analysis in the manner described previously under "Mass Spectrometric Analysis of the Waxes" to determine the highest molecular weight materials present.

Optical Determinations

The distribution of the various waxes in the Comp B matrix was determined under contract by Walter C. McCrone Associates, Inc., Chicago, IL. For such determinations aliquots of the core samples were melted between a slide and a cover slip and observed during crystallization of the TNT. A small hot stage was fashioned so that the slide containing the melted Comp B could be maintained at a temperature of about 82°C during examinations. The sample could also be cooled to approximately 78°C so that slow crystallization of the TNT could proceed. In this manner the wax, as globules, was easily visible before resolidification of the TNT, and was also quite apparent after the TNT was allowed to crystallize just below its melting point. Most importantly, it was observed that the wax did not wet the glass surfaces of the slide and cover slip. Accordingly, this procedure serves as a valid technique for studying the distribution of the waxes in such castings. The hot stage was equipped with a camera to record the distribution of the wax globules in the Comp B matrix.

RESULTS AND DISCUSSION

Characterization of Waxes

The characterization of waxes was reviewed by Robinson and Johnson (ref. 2) who concluded that a combination of column chromatography, gas chromatography (GC) and infrared (IR) spectrometry is required for a complete determination. However, each of these techniques has its limitations. For example, IR analysis is used for the rapid fingerprinting of wax types although similar types such as hydrocarbon waxes in admixtures may be hardly distinguishable. GC has been used by Ludwig (ref.3) to resolve hydrocarbons up to a limited number of carbons and Levy (ref.4), who used mass spectrometry (MS) in conjuntion with GC, quantified hydrocarbons, with various side chains, only up to C_{33}. Column chromatography, at best, is tedious and time consuming with poor resolving capability. More recently,

thermal gravimetric analysis (TGA) and differential thermal analysis (DTA) have been used for the qualitative determination of fractionation of waxes in Comp B (ref. 6); and differential scanning calorimetry (DSC) for the analysis of separated wax fractions in Comp B (ref. 6). For a more direct and complete characterization of waxes, gel permeation chromatography (GPC) appears to have the greatest potential.

GPC was introduced by Moore (ref. 7) as a technique for the examination of high molecular weight substances in solution by an exclusion principle involving high resolution liquid chromatography. With the selection of a Styragel® column packing of appropriate porosity range, most polymeric substances can be examined over the range C_{15}-C_{100}. The effective application of GPC for the characterization of hydrocarbon and ester waxes was reported by Hillman (ref.8). In this study, the GPC technique, checked by MS, was used for the determination of the chain lengths and overall carbon chain ranges comprising the various waxes investigated.

The gel permeation chromatograms of the various waxes considered for incorporation in Comp B, shown in figures 2-14, were obtained by the procedure developed at Waters Associates. The molecular weight distribution of the waxes depicted in figures 2-9 can be divided into two types; samples that have a simple log normal molecular weight distribution and those wax samples with complex molecular weight distributions. The samples with the simple log normal distributions are Standard Wax Knoxville 123, Witco 68097, Sunoco 985B and Petrolite ES670. Those with the complex molecular weight distributions are Indramic 170-C, Sunoco 8810, Sunoco 1290B, and refined yellow beeswax. On each of the chromatograms the molecular weight at various interesting points throughout the molecular weight distribution curve are noted. Thus one can describe each molecular weight distribution group by molecular weight. The Bareco series, figures 10-14, display molecular weight distributions which are log normal in appearance. Most of these waxes cover molecular weights ranging from 500 to 5000. The sample of Bareco X-719, (fig 13) does have a significant high molecular weight tail.

The MS was used to verify the GPC method of determining molecular weight and corresponding number of carbons in the straight-chain hydrocarbons. Several samples of commercial waxes were subjected to fractionation by GPC and fractions corresponding to different but known molecular weight ranges were obtained. Each of these fractions was then analyzed mass spectrometrically and a comparison of the findings is summarized in table 1. Up to a molecular weight of approximately 1100, a qualitative correspondence exists between the molecular weight fractions as determined by GPC and MS. Molecular weights above 1100 are not observed by MS due to volatility limitations. It is reasonable to conclude that each of these samples may contain higher molecular weight fractions.

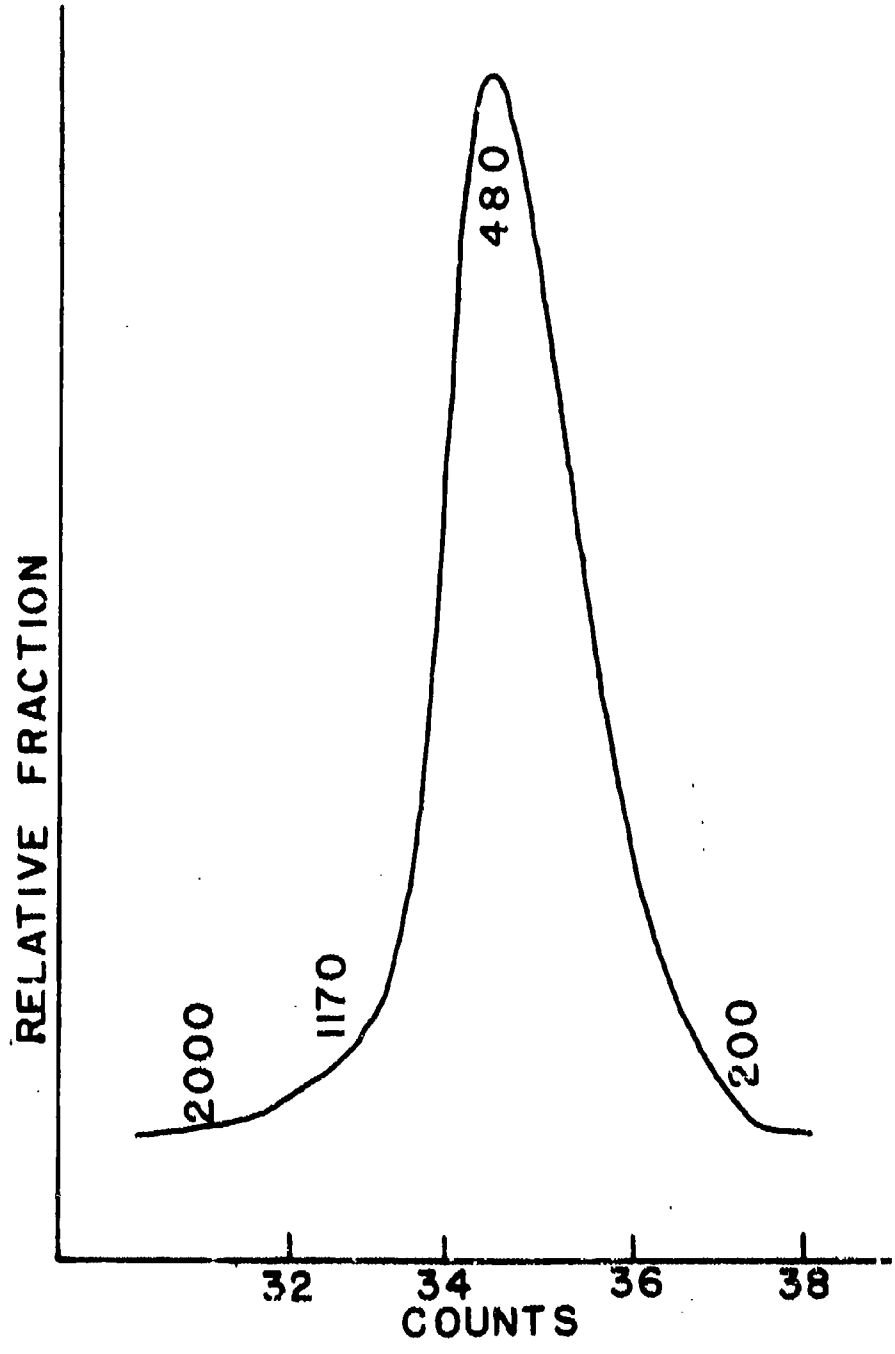

Figure 2. Standard Wax of Knoxville 123.

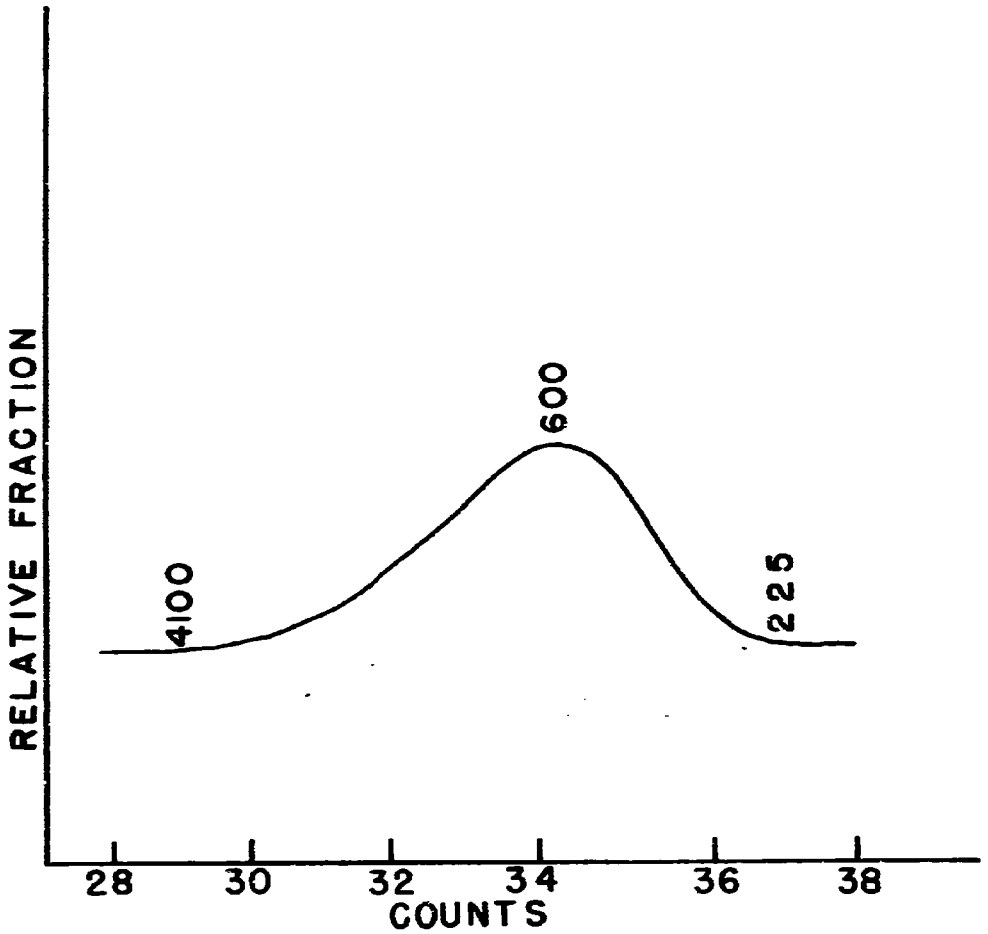

Figure 3. Witco 68097.

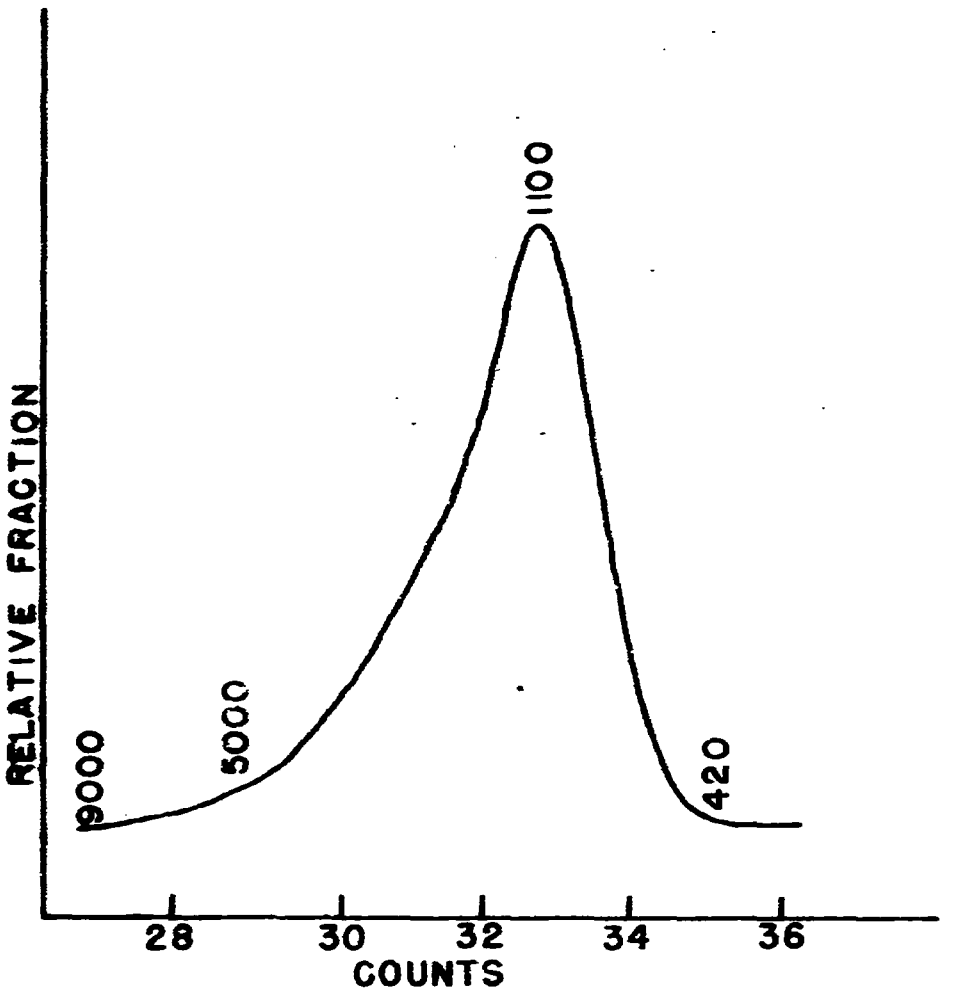

Figure 4. Sunoco 985 B.

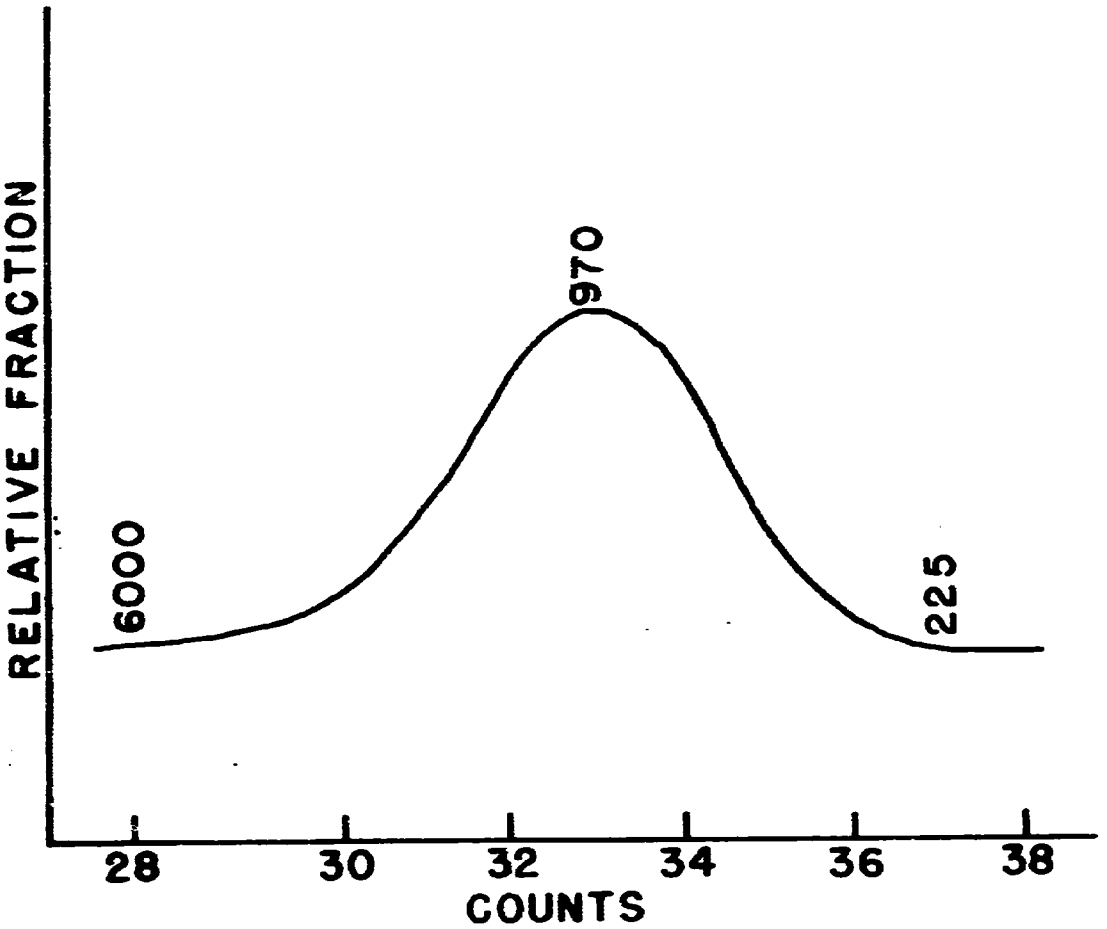

Figure 5. Petrolite ES 670.

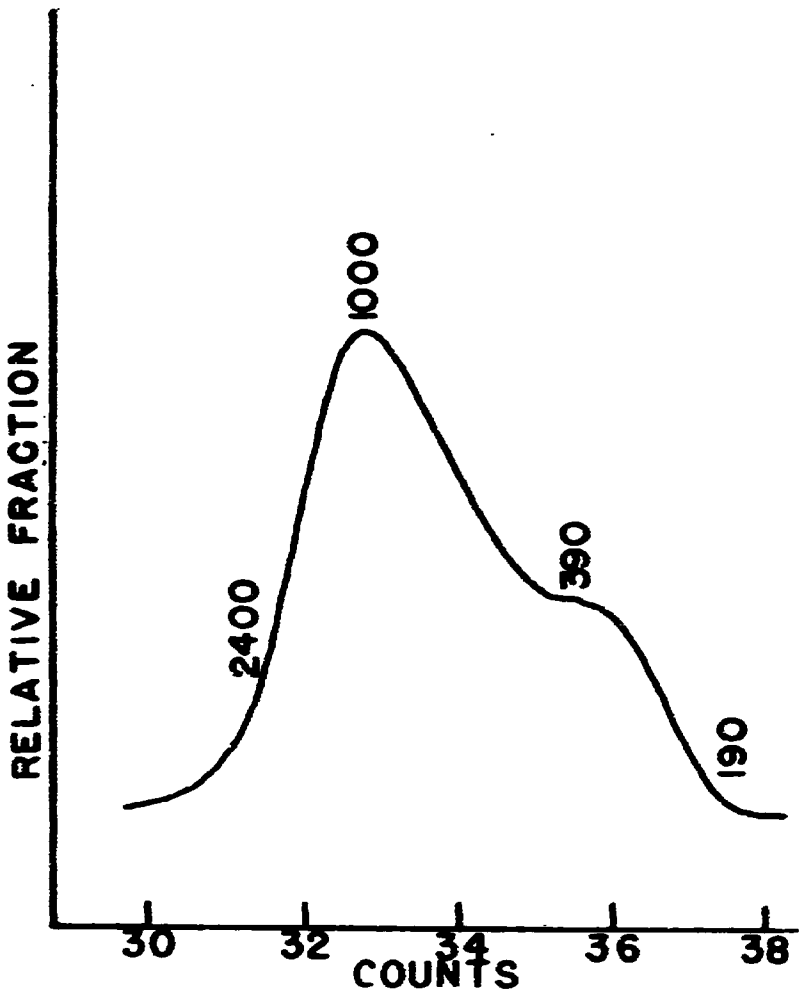

Figure 6. Indramic 170-C.

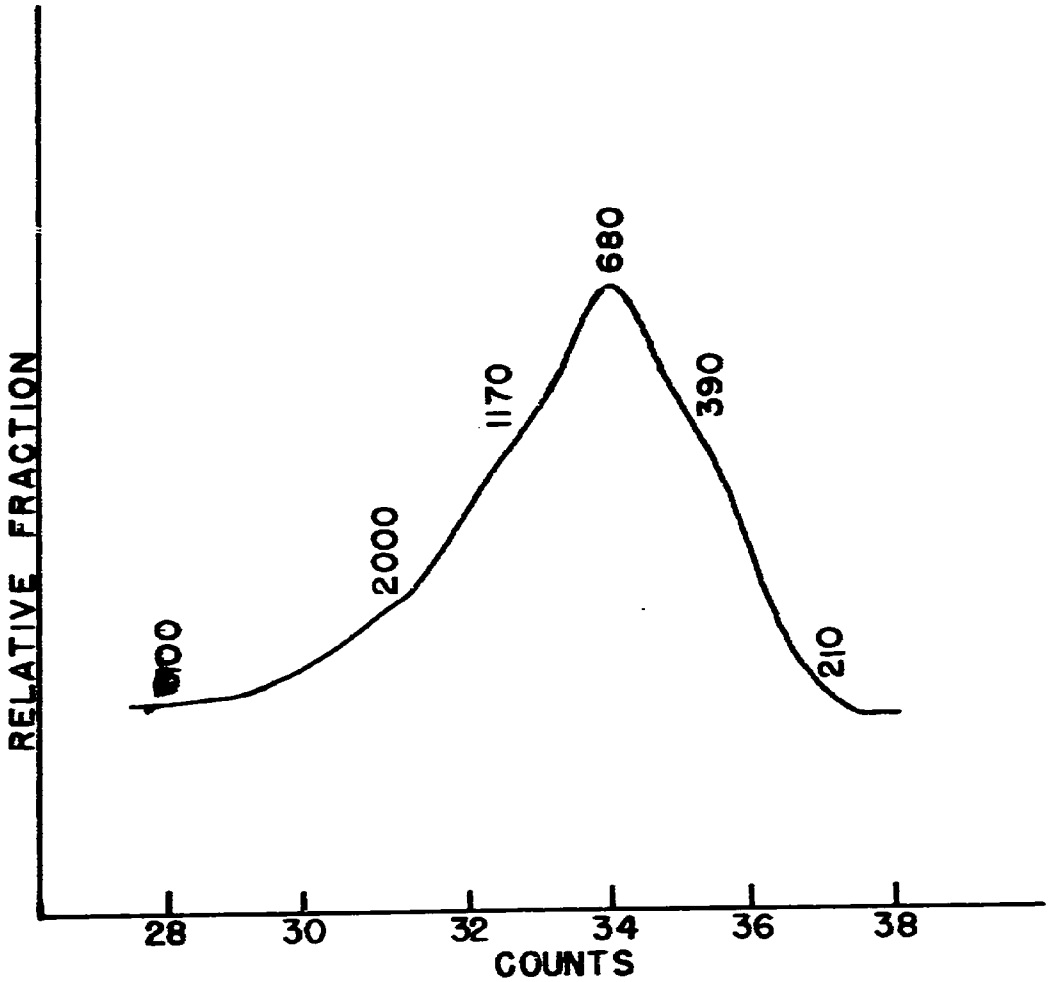

Figure 7. Sunoco 9810.

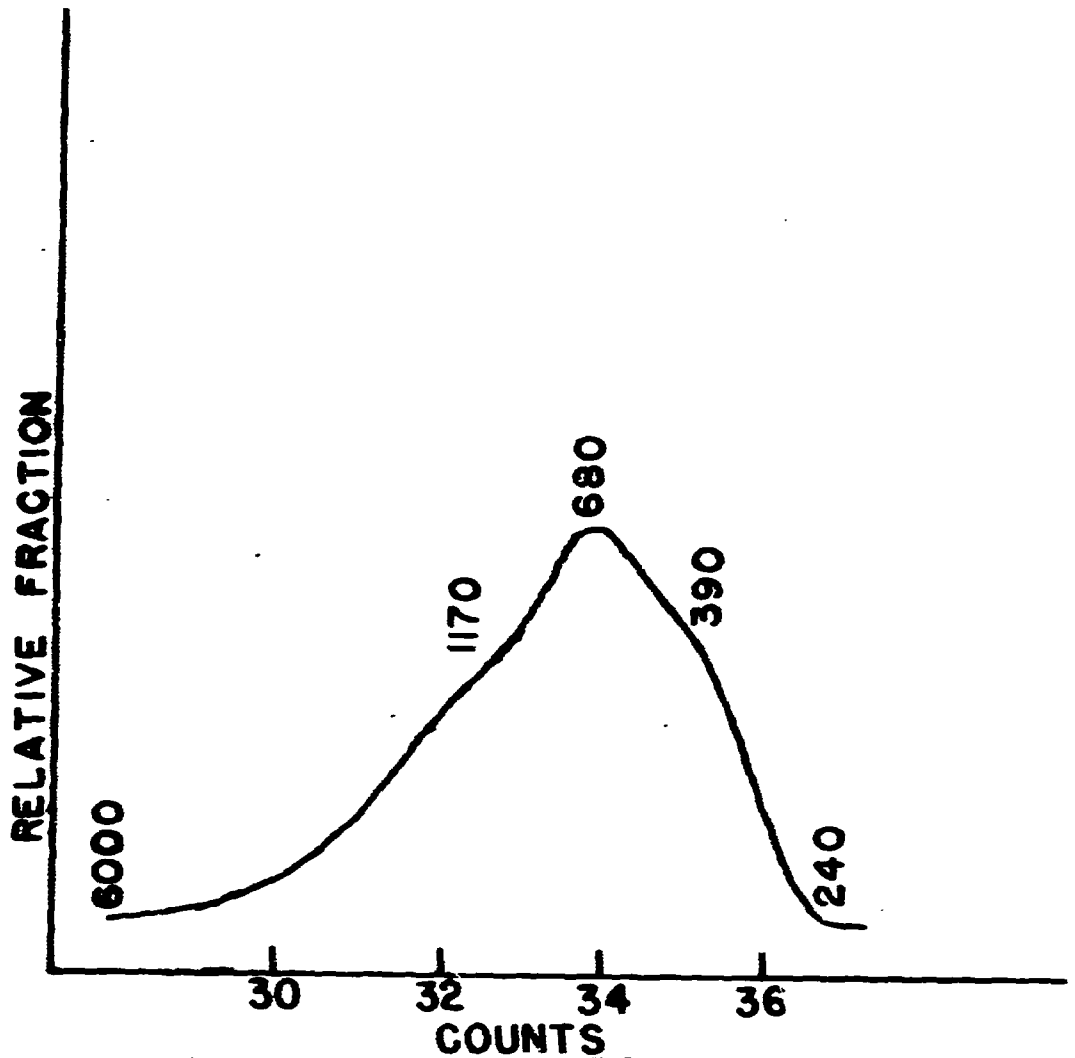

Figure 8. Sunoco 1290B

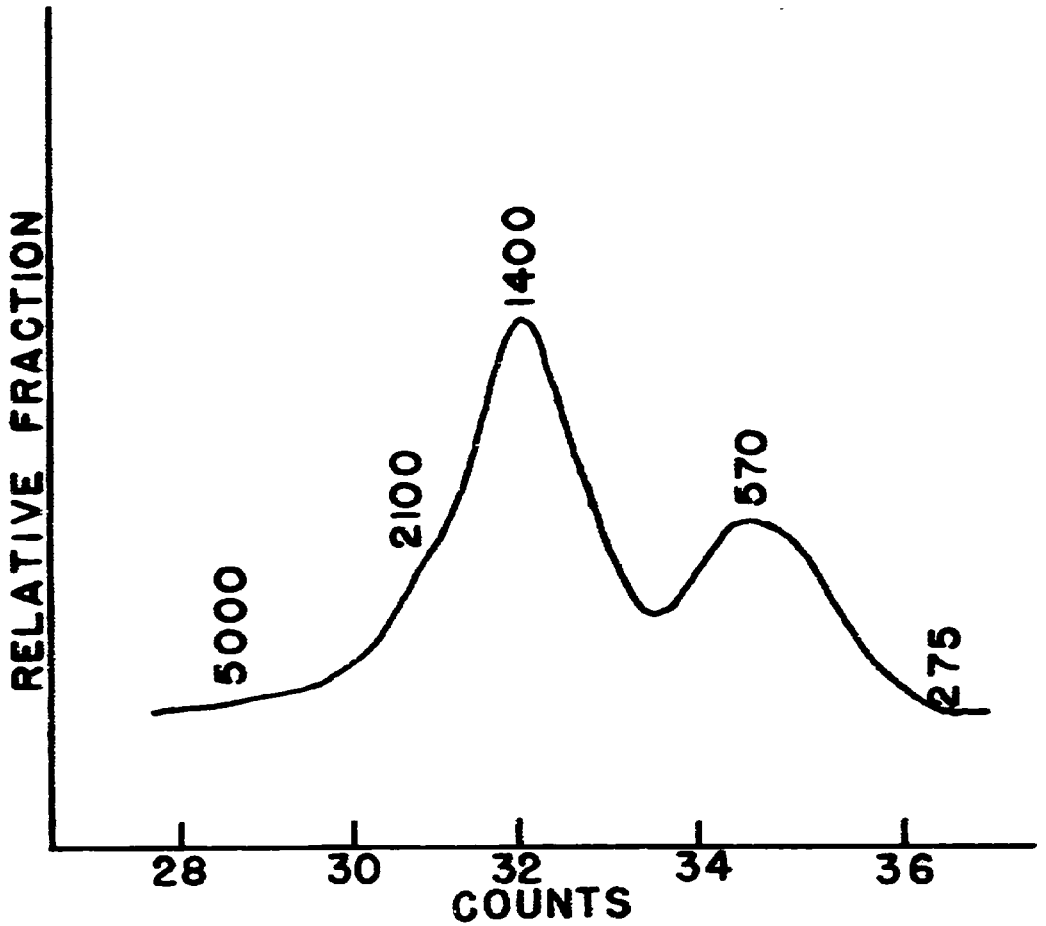

Figure 9. Refined yellow beeswax.

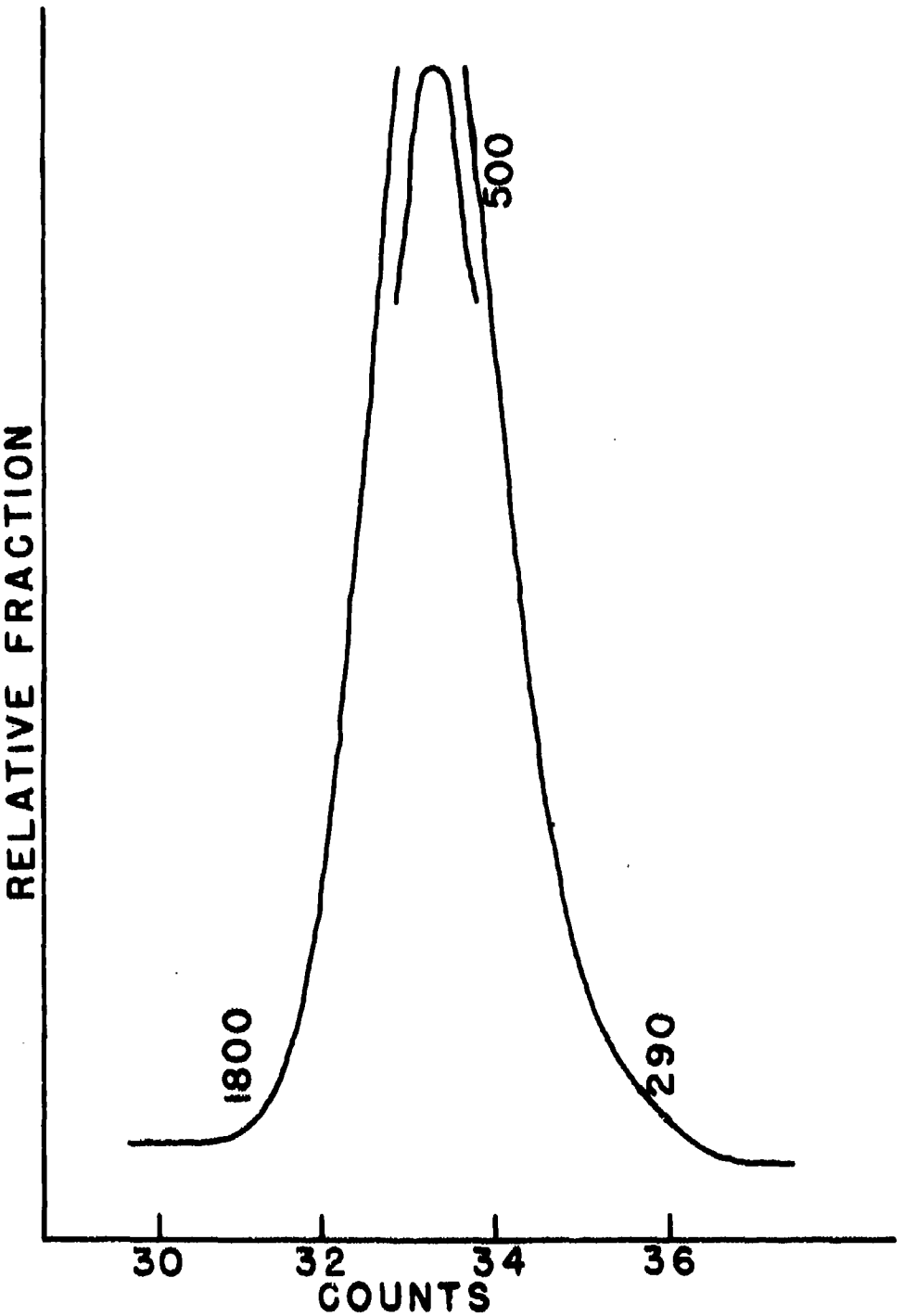

Figure 10. Baraco X-715 Lot C-652.

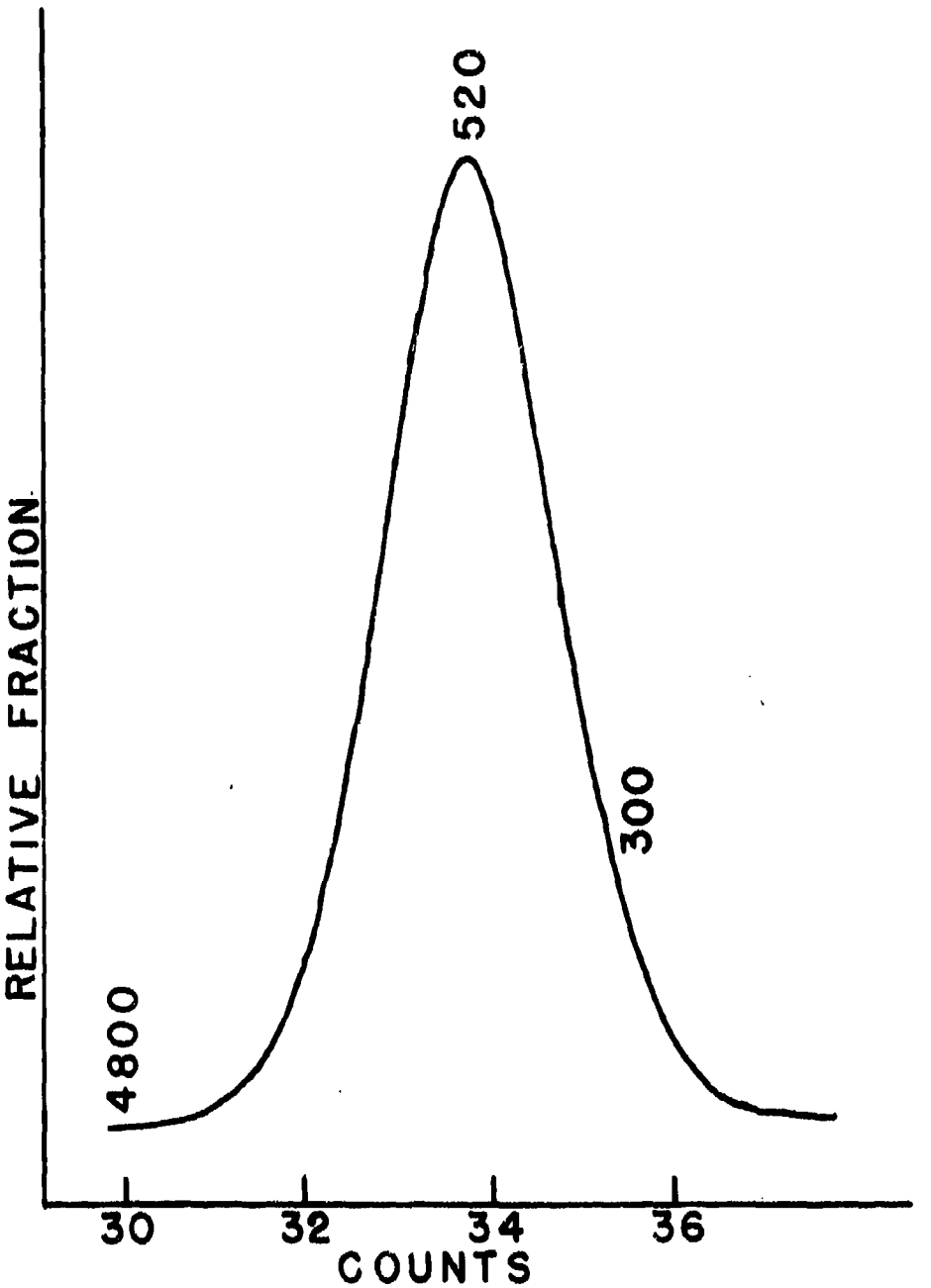

Figure 11. Baraco X-404 Batch B-610.

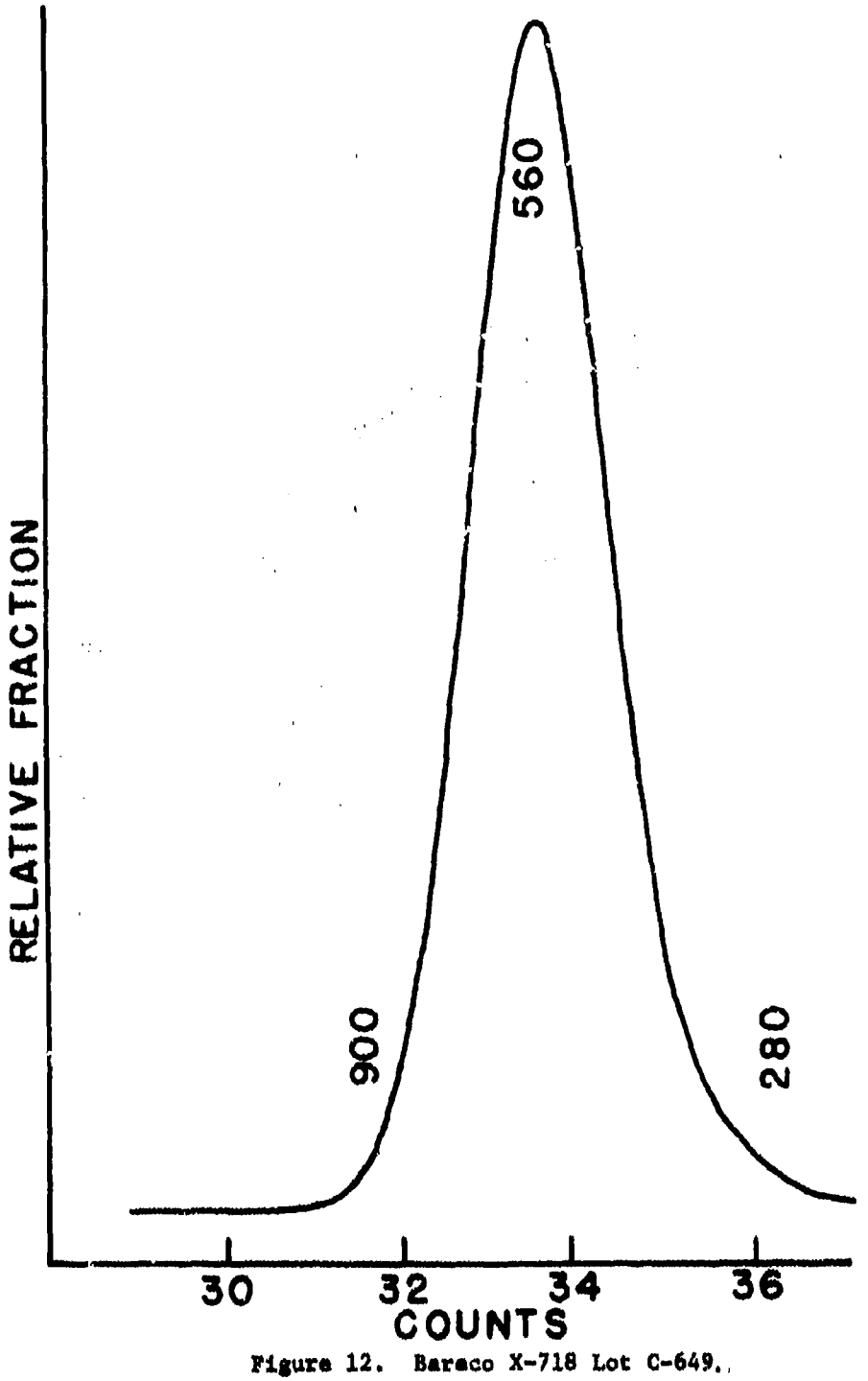

Figure 12. Bareco X-718 Lot C-649.

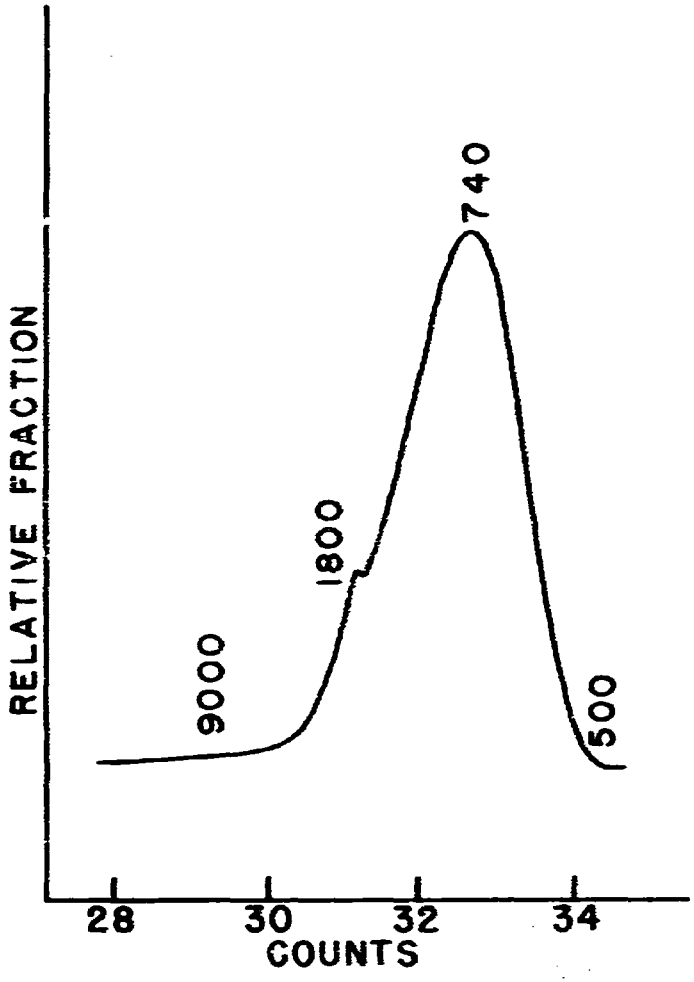

Figure 13. Bareco X-719 Batch 658.

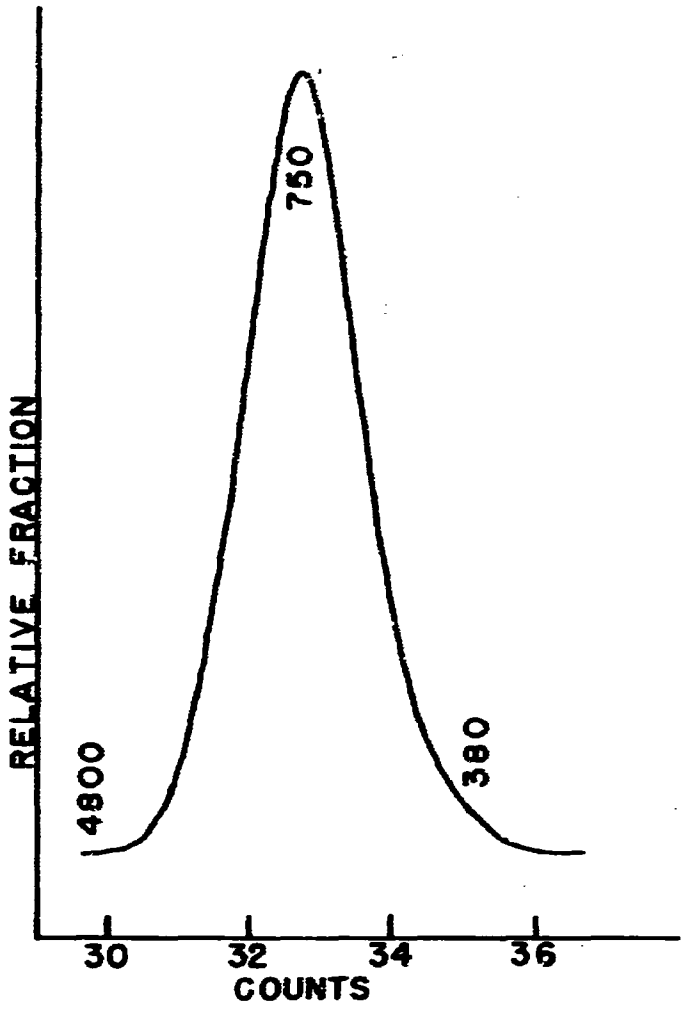

Figure 14. Bareco X-717 Lot C-655.

Table 1. GPC/MS molecular weight determinations of commercial waxes

Wax	GPC Cut # / Fraction #	GPC MW Range Carbon Chain Length	MS MW Range Carbon Chain Length
Indramic - 170	35-36 / 6	400 $C_{28}H_{58}$	520 $C_{37}H_{76}$
Indramic - 170	32-34 / 3	1000 $C_{71}H_{144}$	970 $C_{69}H_{140}$
Petrolite - 670	32-34 / 5+6	800 $C_{57}H_{116}$	725 $C_{51}H_{104}$
Knoxville - 123	35-36 / 3	650 $C_{46}H_{94}$	580 $C_{41}H_{84}$
Sunoco - 985B	25-29 / 2+3	5500 $C_{390}H_{782}$	1100 * $C_{78}H_{158}$

* Maximum Limit of MS Method

Wax Distribution in Comp B Cast

Fractionation

The GPC method for determining molecular weight distribution of the waxes composed of a wide range of chain length molecules could serve to measure the degree of fractionation, if any, that takes place during casting operations. To determine if a poor or good cast was associated with the fractionation of the wax polymer chain lengths, the cast Comp B in two loaded 105 mm projectiles with different physical properties was examined. One projectile was loaded at KAAP with Comp B (K-254 series) formulated with Petrolite ES670 and the other loaded at JAAP with Comp B (J-212 series) compounded with Indramic 170-C. The K-254 cast was deemed acceptable, whereas the J-212 Comp B cast was unacceptable due to cracks and voids. Core samples were removed from the respective casts at the locations indicated in figure 15. In addition to the analysis by the GPC technique, mass spectral analysis was included as a check. The data obtained by the two methods of analysis are summarized in table 2.

Both the number-average molecular weight ($\overline{MN}$) and the weight-average molecular weight ($\overline{MW}$) values of the Petrolite used in the K-254 series of all three core samples as indicated by GPC have not deviated from the corresponding values of the neat Petrolite. In comparison, the maximum m/e ions observed mass spectrometrically in the same three core samples, although below the maximum detectable, are identical within experimental deviation and hence in agreement with the GPC results. For the K-254 series formulated with Petrolite there is no detectable fractionation in the Comp B cast as indicated by the GPC and MS methods. However, in the case of the Indramic wax, the GPC shows skewness of the $\overline{MN}$, and $\overline{MW}$, values to higher fractions (signifying fractionation) for the center peripheral (2) core sample (fig. 15) of the Comp B cast, J-212 series. This observation is supported by the MS analysis, showing a maximum m/e ion value for the same (2) core samples also denoting fractionation (fig. 15). It should be noted that the Comp B formulated with Petrolite, which showed no signs of fractionation, represented the acceptable cast. Whereas the Comp B compounded with Indramic in which fractionation was found contained voids and cracks. This association of fractionation with the quality of casting, as revealed by GPC analysis, demonstrates possibly that the GPC technique is capable of detecting changes in the wax composition which bears some relationship to the physical properties of cast Comp B. What should be done is to establish this capability on a statistical basis, and then proceed to determine what the relationship is by a detailed parametric study.

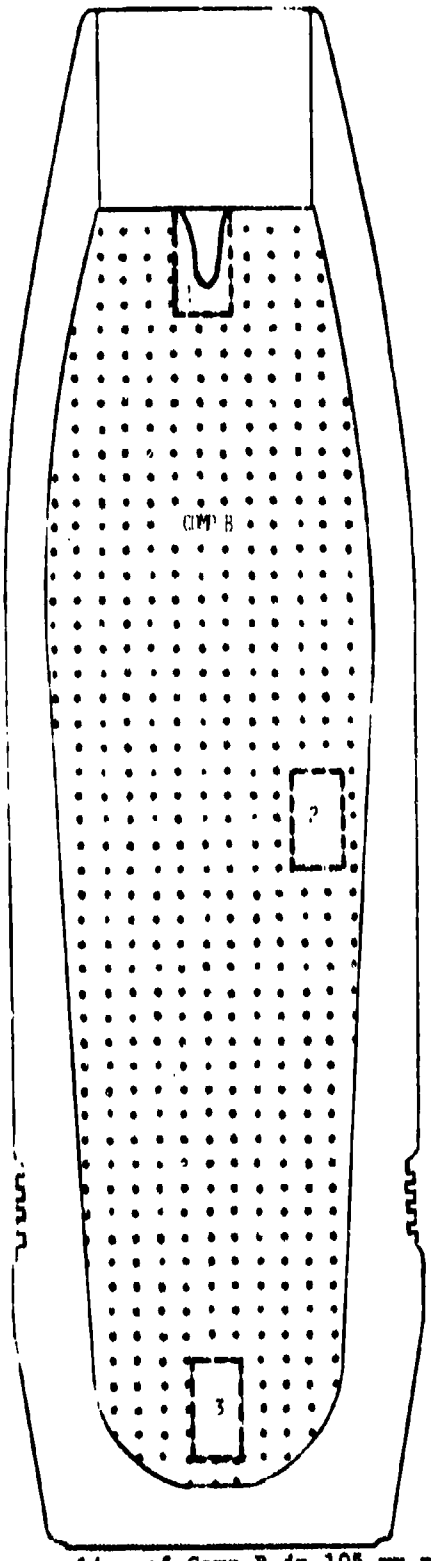

Figure 15. Core sampling of Comp B in 105 mm projectile.

Table 2. Fractionation of waxes in Comp B casts.

Wax	GPC Analysis $\overline{MN}$			GPC Analysis $\overline{MW}$			Mass Spec Analysis Max m/e ion		
	1	2	3	1	2	3	1	2	3
Standard		(970)[a]			(1250)[a]				
Petrolite ES670									
In Comp B Cast K-254 Series	960	1050	1010	1040	1150	1090	965	1020	980
Standard		(1000)[a]			(1110)[a]				
Indramic 170-c									
In Comp B Case J-212 Series	980	1329	980	1070	1445	1020	780	1075	910

1. Top center
2. Center peripheral
3. Bottom center (core samples of Comp B cast)

a Neat wax

Dispersion

Figure 16 shows a representative field with rounded globules of wax distributed throughout the continuous TNT melt. The diameters of these globules range from barely visible at this magnification, perhaps 2 to 3 micrometers in diameter, up to about 150 micrometers. The globules are lighter than the TNT melt and tend to float to the top where they lie, primarily, in a single plane making photomicrography much easier. Figure 17 is a different field of the same preparation shown in figure 16, simply to show the variation from one area to another. The number and size distribution of wax globules appear to be essentially the same in the two views. These two figures show material taken from the sawed surface of the sample. Figure 18 is the same field of view as in figure 17 after cooling a few degrees to allow the TNT to solidify. The wax globules can still be seen and are only slightly distorted by the TNT crystal growth. Finally, figure 19 shows the same field of view as in figures 17 and 18 and the preparation has been reheated to remelt the TNT. Its appearance is practically identical to figure 17 which was taken before the TNT had crystallized and been remelted. Close examination shows minor displacements of the wax globules but no significant change in shape, size or dispersion of any of the phases. As a result, it is concluded that the way the wax globules appear in the melt is representative of the way that the wax will exist in the cast product.

Figure 20 is a sample taken from the inside surface of a large cavity in the same casting, whereas the sample used for figure 16 was taken from the sawed surface of the casting. If anything, there seems to be less wax in this sample than in the material shown in figures 16-19. In scanning the two samples there seemed to be no significant difference in amount or distribution of wax. Figure 21 shows the sample may well contain more wax than any of the others. This figure shows three wax globules exceeding 100 micrometers in diameter and a considerable number of smaller globules are also easily visible. This view was chosen to show the not uncommon occurrence of several large globules in a specimen. Figure 22 is another view of the same sample shown in figure 21 and was chosen to show the large number of very small wax globules in this area. The desensitizing effect of the wax could be greatly affected by the size distribution, but it was not possible, within the scope of this work, to accurately measure the size distribution.

Figure 23 shows that this sample is similar in all respects to J-212 1. Figure 24 shows that this sample possibly contains less wax than some of the others but not significantly so. Figure 25 is a representative field showing little variation in amount or size distribution from most of the other samples. Figure 26 appears to indicate that this sample may contain more wax than some of the others.

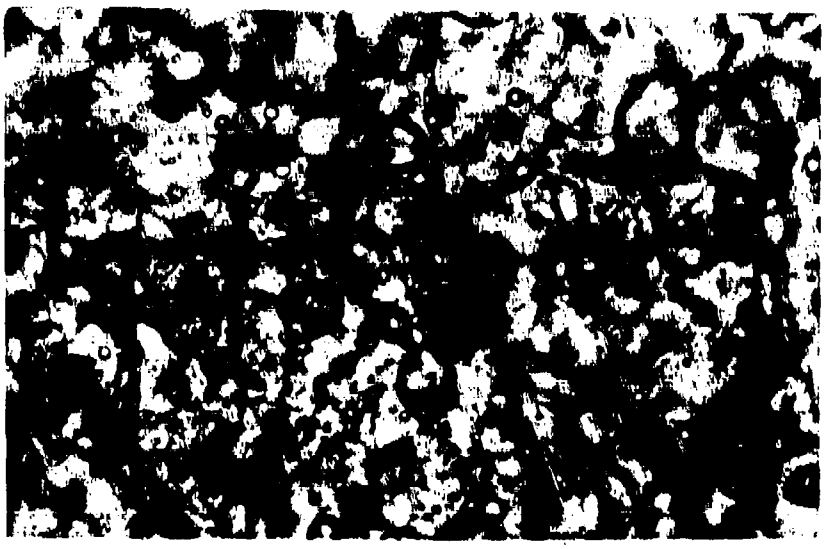

Figure 16. J-212 1: Indramic 170-C dispersion in Comp B cast core sample 1 taken from top center.

Figure 17. J-212 1: Indramic 170-C dispersion in different field of sample preparation shown in Fig 16.

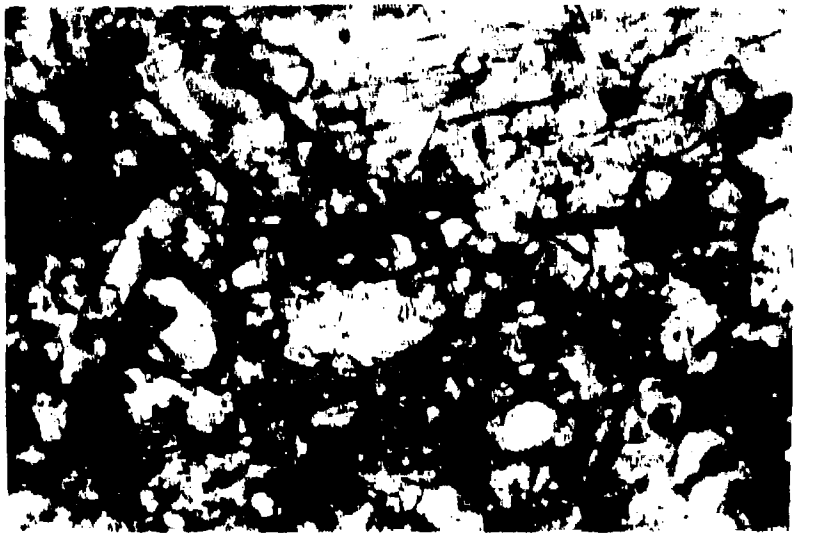

Figure 18. J-2121: Same field of view as in Fig 17 after solidification of TNT.

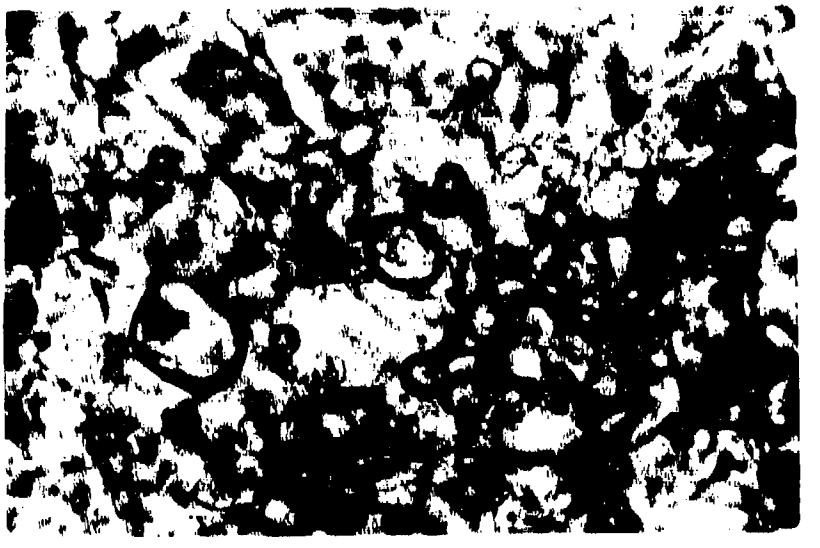

Figure 19. J-2121: Same field of view as in Fig 18 after remelting TNT.

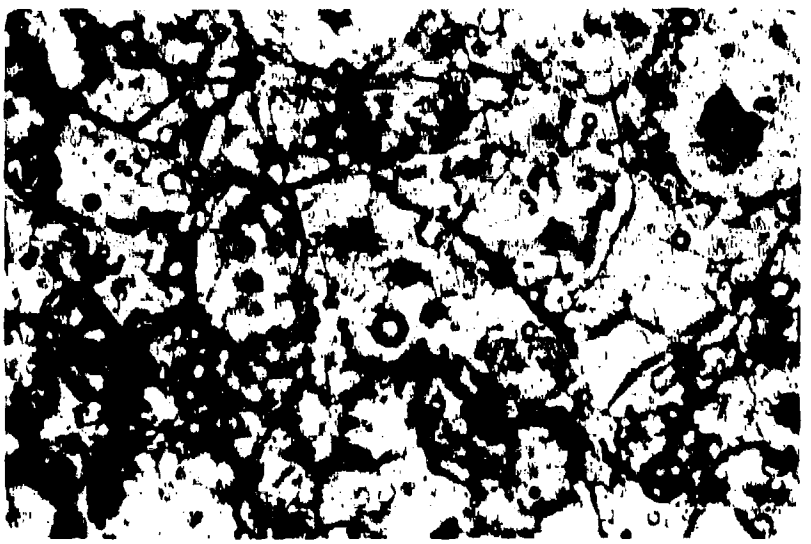

Figure 20. J-2121: Different preparation of sample 1.

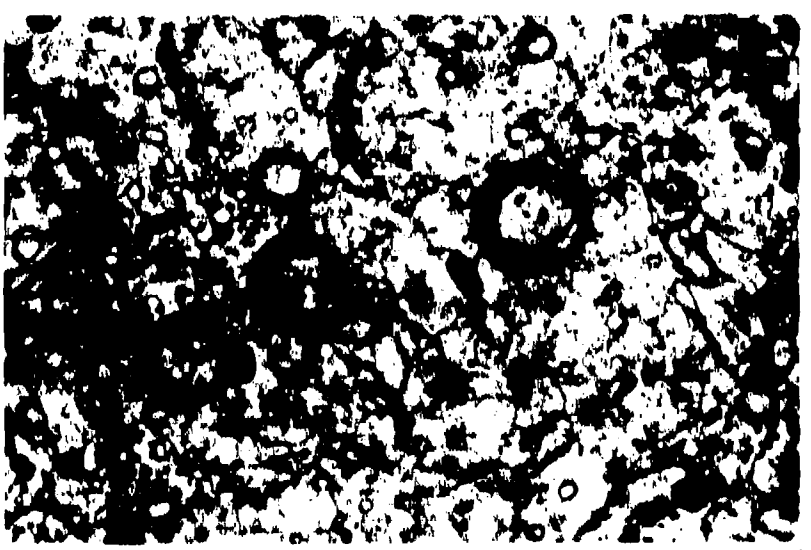

Figure 21. J-2122: Indramic 170-C dispersion in Comp B cast core sample 2 taken from center periphery.

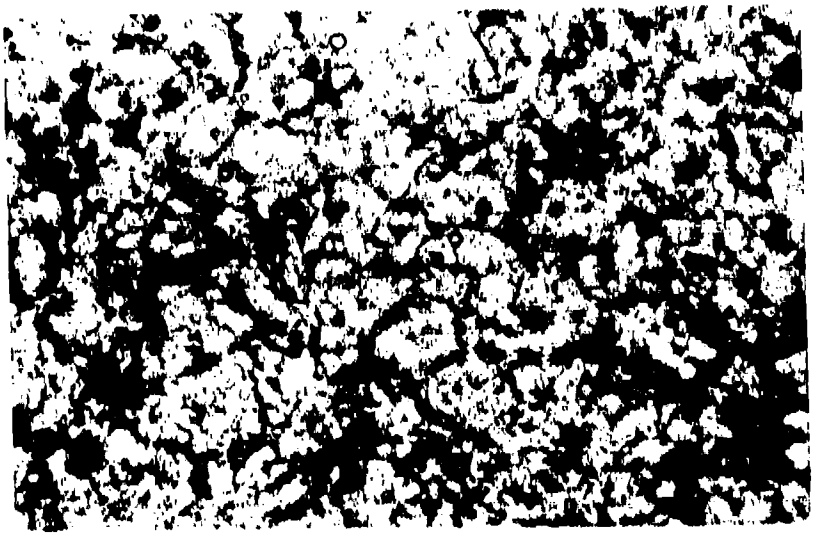

Figure 22. J-2122: Different field of view of same sample preparation in Fig 21.

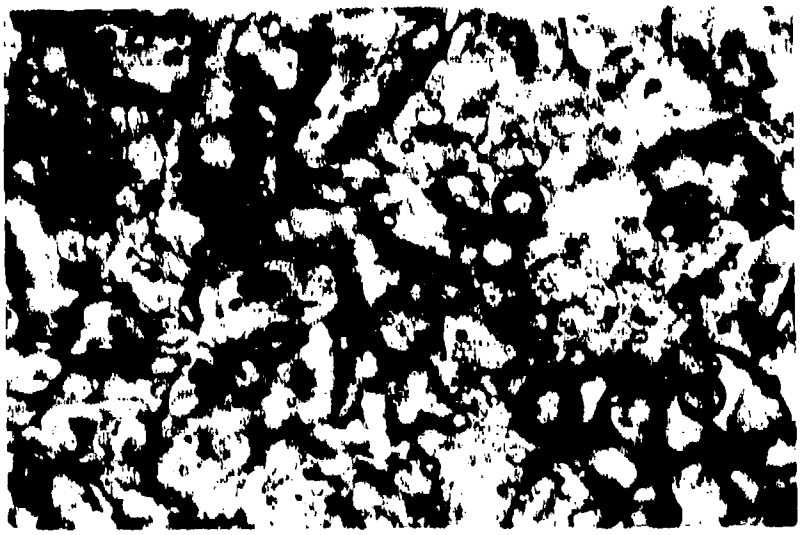

Figure 23. J-212 3: Indramic 170-C dispersion in Comp B cast core sample 3 taken from bottom center.

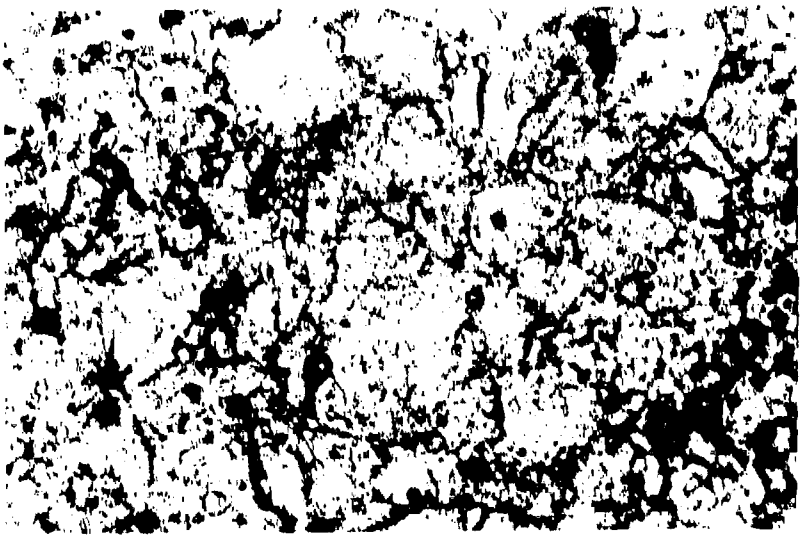

Figure 24. K-254 1: Petrolite ES670 dispersion in Comp B cast core sample 1, taken from top center.

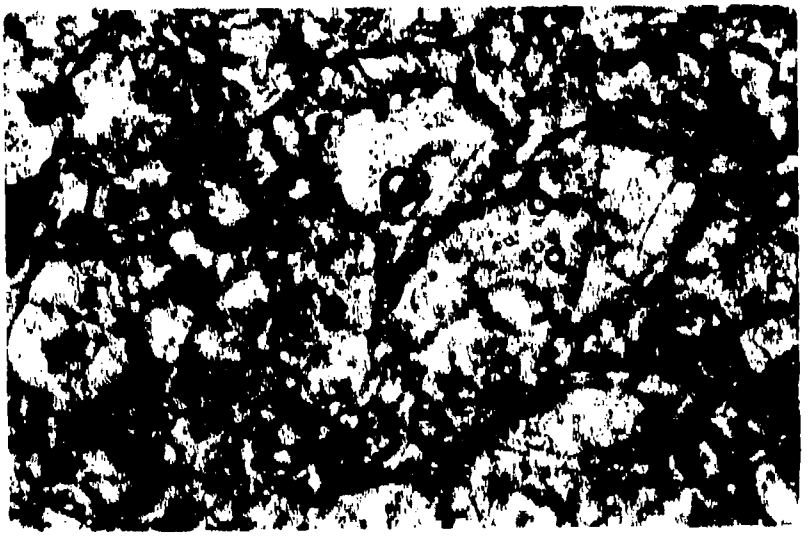

Figure 25. K-254 2: Petrolite ES670 dispersion in Comp B cast core sample 2, taken from center periphery.

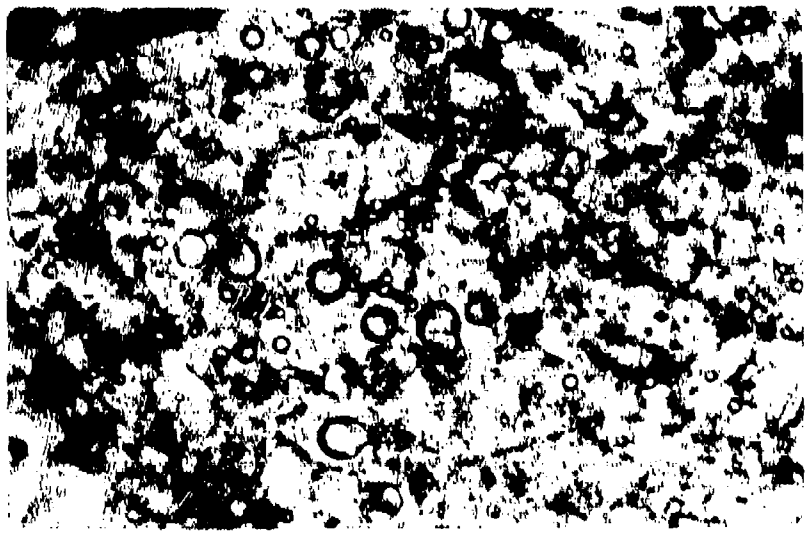

Figure 26. K-254 3: Petrolite ES670 dispersion in Comp B cast core sample 3, taken from bottom center.

Figure 27. K-254 2: Different preparation of sample 2 showing impurities wetted by wax.

Figure 27 shows one oak leaf hair (a trichome) that appears to be easily wetted by the wax. In every case, the wax globules seem only to wet paper fibers and other biological materials that happen to be present as contaminants in the casting. We would expect that wax would do a much better job of desensitizing if it did wet the RDX crystals or at least the growing TNT crystals. This is obviously not the case with these waxes. Once again it appears that the behavior of the Indramic wax in the center peripheral core sample of the Comp B cast is different from that of the Petrolite wax. In this case the difference is based on the dispersibility of the wax in the Comp B matrix. The Indramic wax is observed to be dispersed in large globules, whereas the Petrolite globules are finely subdivided and uniformly dispersed.

Wettability

To determine differences in the wettability among the various waxes, a study of the contact angle of the molten waxes on RDX was undertaken. The wettability of a solid surface by a liquid may be estimated by means of the contact angle (ref. 9). The contact angle gives information about wettability and also, indirectly, about surface energetics and heterogeneity. An equilibrium drop, in contact with a surface, will take the shape which minimizes the free energy of the system. The angle which a drop assumes on a solid surface is the result of a balance between the cohesive forces in the liquid and the adhesive forces between solid and liquid. If there were no interaction between liquid and solid, the contact angle would be 180°. When the interaction is sufficient, the liquid can spread (contact angle = 0°); it follows that there are intermediate situations where the contact angle lies between these two extremes.

To verify the techniques for the determination of contact angles, the contact angle of water and molten TNT on RDX was measured. The results were as follows:

	Contact angle, (Degrees)		
	Water on RDX	TNT on RDX	TNT on HMX
This work	67	22	-
Eadie (ref. 10)	69	-	28
Evans (ref. 11)	65	24	-

The results from using the technique described in this work agree rather closely with previously reported work.

The contact angles of molten waxes, Witco, Indramic, Petrolite, Sunoco 8810, Sunoco 1290B, Sunoco 985B and Standard Wax Knoxville 123, as well as molten TNT, on the RDX surface were found to be either zero or close enough to zero to indicate that spreading took place in every case. The experiments were repeated in a water-saturated atmosphere with the same results. Thus, differences among the various waxes could not be distinguished.

Since all waxes on contact with RDX spread, it was possible that the contact angles of these waxes measured on glass could show up differences among the waxes. The results were as follows:

	Contact angle, (Degrees)	
Wax	Ambient Atmosphere	Water Sat'd Atmosphere
Sunoco 8810	18	21
Sunoco 985B	22	22
Sunoco 1290B	23	25
Indramic 170C	21	21
Witco 68097	23	20
Petrolite ES 570	26	20
Knoxville #123	19	22

As can be seen there are no significant differences among the waxes, so far as their contact angles on glass are concerned.

Additional contact angle measurements were made to show that molten TNT displaces all the waxes used in these experiments. This is in agreement with Eadie (ref. 10) and with other investigators (refs. 1 and 12) who point out that the contact angle of melted high explosives against most solid, dispersible organic booster explosives is small or zero.

As a further check on the wettability of RDX by wax and TNT, some small laboratory batches of Composition B were prepared using a different wax for each batch. Each batch was kept molten for one hour and then allowed to cool. In these experiments most of the wax appeared to remain dispersed in the Comp B mixture. The small size of these batches, however, ensured rapid cooling upon removal from the steam bath, and thus are not realistic, when compared to plant batches. Again, the RDX was completely wetted by the TNT and the wax appeared to be displaced by the TNT. The experiments did show that the wax can be kept emulsified or spread into small droplets. It has been shown (ref. 13) and verified in this study that the wax in Comp B exists as tiny droplets distributed throughout the explosive and not as a coating on the RDX crystals.

Solubility

Consideration has been given to the possibility that the solubility of the waxes in TNT could play a significant role in the pouring quality of the Comp B mixture and/or in the sensitivity of the cast. The waxes cited in this study were tested for solubility in TNT and all were found to be insoluble by the method used. Most of the wax, approximately 70%, migrated to the top and the remainder to the sides.

CONCLUSIONS

The GPC method of analysis shows a potential capability for determining the degree of fractionation of polymeric hydrocarbons of varying carbon chain lengths. Once established, the technique could be applied to determine a relationship, if any, between fractionation and physical and mechanical properties of the Comp B cast. The various waxes tested exhibit no difference in their wettability of the RDX surface, nor in their insolubility in molten TNT. TNT preferentially wets the surface of RDX and disperses the wax as globules throughout the TNT/RDX Comp B matrix. The dispersed globules of wax vary in their size and distribution in the Comp B cast. It is possible that the various waxes may differ in their degree of fractionation, emulsifiability and homogeneity of dispersion in the TNT/RDX matrix. All of these factors may, in part or in combination, contribute to the variations observed in the physical and sensitivity properties of the Comp B cast.

The results cited in this investigation on the behavior of wax in Comp B, some of which repeat and confirm previous work, point to the need for fundamental data. For lack of such basic information, the role of wax in Comp B is still unresolved after many years of effort. Only preliminary fundamental studies have been reported (ref. 14) on the surface energetics of some of the organic secondary explosives. Basic data on the cohesive, adhesive and electrostatic forces of adsorbate-substrate interaction are required before any effective approach to the coating of explosives can be achieved.

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